RESEARCH CIRCULAR 88 AUGUST 1960

THE MANUFACTURE OF APPLE SIRUP

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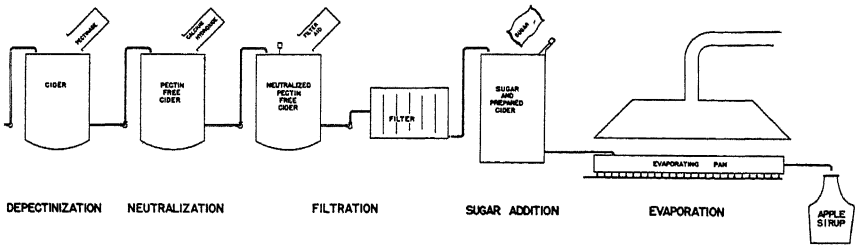

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SUMMARY—A schematic diagram of the steps involved is shown on the cover. There are three tanks shown to represent the three steps of depectinization, neutralization, and filter aid addition, but all three of these steps could be carried out in only one tank. Three individual tanks are not necessary.

By way of summary, a step by step example follows:

1. Have 150 gallons apple juice available.
2. Want to add sugar to produce an apple juice with 40% sugar before evaporation begins.
3. Desire a medium tart sirup so neutralize apple juice to 0.30% acid.
4. The acidity of the juice is determined as directed on pages 4 and 5. Found it took 7.8 ml. sodium hydroxide.
5. In Table 2 across from 7.8 ml., the acidity of the juice is found to be .5226%. Under the column headed 0.30% and across on the same line from 7.8 ml., it is found that 1 pound 1 ounce of calcium hydroxide is required to neutralize the correct amount of acid in 100 gallons of apple juice. However, it is desired to neutralize 150 gallons of juice so this figure must be multiplied by 1.5 (for 200 gallons the multiplier would be 2). The result is 1 pound 9½ ounces. This amount of calcium hydroxide must be added to the 150 gallons of apple juice.
6. For depectinization it is decided to allow the juice to stand over night which will amount to about 15 hours time. The temperature of the juice is 40° F. In Table 1 under 15 hours and across from 40° F., it is found that 30 ounces of pectinal-A are required for 100 gallons. Since there are 150 gallons, the 30 ounces are multiplied by 1.5 and the answer is 45 ounces of pectinal-A should be added to the juice.
7. After settling the juice is filtered by use of a filter aid and filter.
8. By use of a hydrometer calibrated in degrees brix, we find that the juice contains 11.5% sugar. It was decided to add sugar to increase this to 40% sugar. In Table 3 across from 11.5 and under 40%, we find that it will require 412 pounds of granulated sugar to do this.
9. Evaporate by boiling to 70% sugar.
10. Fill into containers at a temperature of 180 to 190° F.
11. Seal the containers and cool rapidly.

THE MANUFACTURE OF APPLE SIRUP[1]

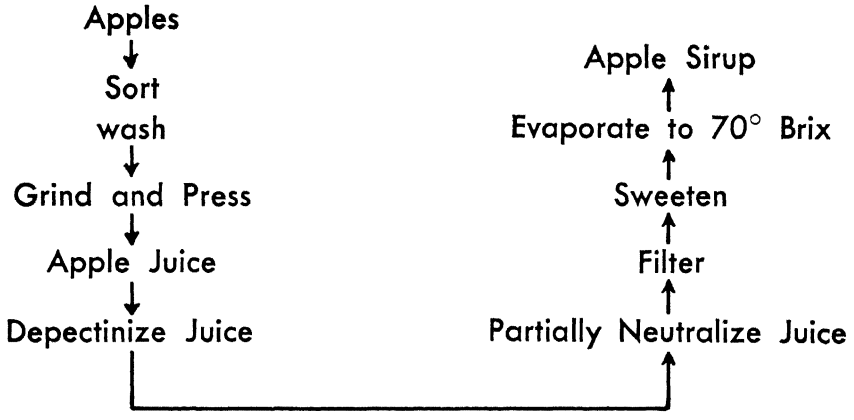
MILTON P. BALDAUF[2]

Apple sirup as a table and cooking sirup is not new as a method for its production was first set forth by H. C. Gore of the U. S. Department of Agriculture in 1914. There have been modifications and refinements made to the original method down through the years. This bulletin describes modifications of the original method to produce a high quality apple sirup.

Another type of apple sirup which is produced is the industrial or humectant sirup. A great deal of this was produced during World War II to replace glycerin as a humectant in cigarets and tobacco. The taste of this product is very bland with a bitter after taste.

The apple sirup produced by the method contained herein has a pleasant apple flavor and a degree of tanginess to give it character. The tanginess or acidity can be varied to suit individual tastes.

The production of apple sirup for table and cooking use is not difficult and does not require a large amount of equipment. Apple sirup is simply the concentrated product of a solution of depectinized, partially neutralized apple juice and sugar. The flow sheet, or steps in the production of the manufacture of apple sirup is as follows:



There are certain precautions, methods, and techniques that need to be followed and they are given in the following sections: (The part of the flow sheet on the production of apple juice is well known and will not be explained here. The first operation to be explained will be the depectinization of apple juice.)

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[1]Recognition is given to D. R. Davis for his assistance in the final preparation of the manuscript and under whose direction further work with this product is continuing.

[2]Formerly Assistant Professor, Department of Horticulture, Ohio Agricultural Experiment Station.

DEPECTINIZATION—Apple juice contains pectin, one of the ingredients necessary to produce a jelly. In order to prevent the formation of jelly during the concentration of the juice, the pectin is eliminated by the use of an enzyme preparation.[3] If the enzymatic preparation pectinal-A is used, the amount to be added can be calculated from Table 1. The data in Table 1 indicates that the amount of enzyme, temperature, and time of action are all interrelated. For example, if the cider is to be left over night, there will be about 15 hours of reaction time. Looking in the column for 15 hours, the amount of enzyme to use will be found to vary greatly with the temperature of the apple juice. The lower the temperature and the shorter the time for action, the more enzyme that must be used. Fermentation, however, must also be considered, thus generally a low temperature and a short time are preferred.

TABLE 1.—Ounces of Pectinal-A to be added to 100 gallons of apple juice to break down the pectin under various conditions of time and temperature

Temperature of juice in degrees fahrenheit	Reaction Time			
	5 hours	15 hours	30 hours	48 hours
40	--	30	15	10
60	54	18	9	6
100	14	5	--	--
120	7	--	--	--

NEUTRALIZATION—Apple juice contains acid which gives it tartness and tang which makes it desirable as a drink. When the juice is boiled to concentrate the sugar, the acid is also concentrated. The concentrated acid is so strong as to make the product too tart for consumption. Therefore, a portion of the acid must be neutralized. In order to reduce the acidity of the apple juice to a known level, the total acidity must be determined. This can be accomplished as illustrated in the series of pictures on pages 6, 7, 8. To find the total acidity, read down column one of Table 2 until the number of milliliters of 0.1N

[3]There are several preparations on the market. The one that was used in these studies was Pectinal-A. It is manufactured by Rohm and Haas Co., Washington Square, Philadelphia 5, Pa.

sodium hydroxide that were used in the titration is reached; across from this number in column two is the percent of acid. The other columns give the weight of calcium hydroxide to use to bring the acidity down to the percent acidity given at the heading of the various columns. The desired level of acidity will depend upon the amount of sugar to be added and the desired tartness of the apple sirup. In most instances decreasing the acidity to .30% produced a most desirable product.

For example, if 6.8 milliliters (ml.) 0.1N NaOH were used, the acidity of the juice is .4556%. If an acidity of .30% is desired, add 12 ounces calcium hydroxide to the apple juice.

Purified calcium hydroxide (very low magnesium content) should be used. It should first be made into a very smooth paste with a small amount of apple juice. The paste should then be dissolved or dispersed into a larger quantity of one to five gallons of warm juice. This juice is then mixed well with the original batch of apple juice. Calcium hydroxide may be added to the apple juice at the same time as the pectinase enzyme or it may be added after the enzyme action is completed.

FILTRATION—The addition of pectinase to the apple juice causes a flocculant precipitate to form. Also, if a calcium hydroxide with a high content of magnesium is used, a granular precipitate will form. Both of these precipitates should be removed from the apple juice before adding sugar and boiling down. It is accomplished by filtering with the use of a filter aid[4] and filter such as is used for apple juice and cider. A fairly retentive grade of filter aid is preferred in order to produce a clear sirup.

ADDITION OF SUGAR—The addition of sugar to the apple juice is optional, but some additional sugar should be used. If no additional sugar is used, the sirup will require a longer time to boil down, become too dark in color, and have an off flavor. Also, if sugar is added, the acidity has to be lowered to such a point so that the sucrose is not inverted and sugar crystalization occurs in the finished sirup. The amount of added sugar will depend upon the relative cost of sugar and heat for evaporation and the type label desired. The more sugar added, the larger the initial volume of juice, and the less heat that is needed to reach the desired concentration. Also the amount of sugar

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[4]Filter aid is generally a diatomaceous silica which is produced in various grades. Two producers of diatomaceous silica filter aids are (1) Johns-Manville Corp., Celite Division, 45 Prospect Ave., N. W., Cleveland 15, Ohio and (2) Great Lakes Carbon Corp., Dicalite Division, 19140 Detroit Road, Cleveland 16, Ohio.

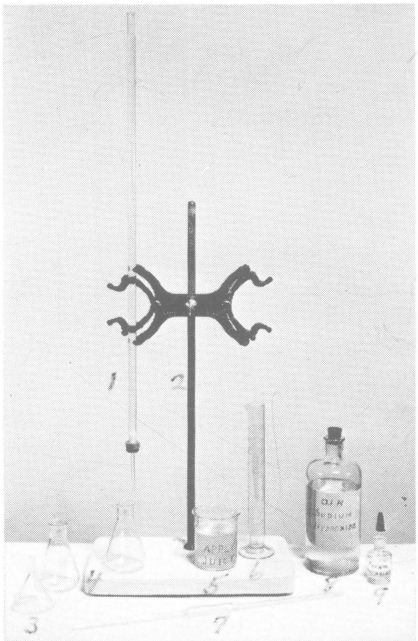

Figure 1

The following equipment is needed and can be obtained from a chemical supply house[5] for about $25.

1. 50 milliliter (ml.) burette
2. burette stand and clamp
3. burette funnel
4. two 125 ml. Erlemeyer flasks
5. any kind of glass container
6. a graduate, 50 or 100 ml. size
7. 10 ml. pipette
8. standard sodium hydroxide solution, 0.1 normal
9. phenolphthalein indicator, 0.02 %

Fig. 1.—The burette (1) is secured by the clamp as indicated in figure 1, and filled with 0.1 N. sodium hydroxide (8) using the burette funnel (3). Make sure the tip of the burette below the valve is full by allowing some of the liquid to run through.

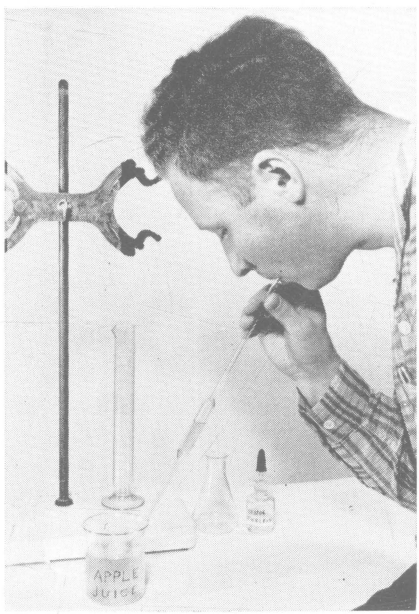

Figure 2

Fig. 2.—Mix the batch of apple juice well and fill a small glass container (5). To take an exact volume of sample for a determination, a 10 ml. pipette (7) is used. The pipette is inserted into the apple juice and the sample is drawn into the pipette as shown in figure 2. The juice is drawn up past the mark on the upper stem of the pipette. The mouth is removed and the top of the pipette is quickly covered with the index finger as shown in figure 3. While holding the pipette in this manner slightly loosen the finger until the juice drips out the bottom. When the level of the juice reaches the mark on the upper stem of the pipette, increase the pressure of the index finger on the pipette so that the flow of juice will stop.

[5]There are many chemical supply houses. Two in Ohio are: (1) Harshaw Scientific Co., 1945 E. 97th St., Cleveland 6, Ohio or 6265 Wiehe Road, Cincinnati 13, Ohio (2) Kauffman-Lattimer Co., 230 North Front St., Columbus 16, Ohio.

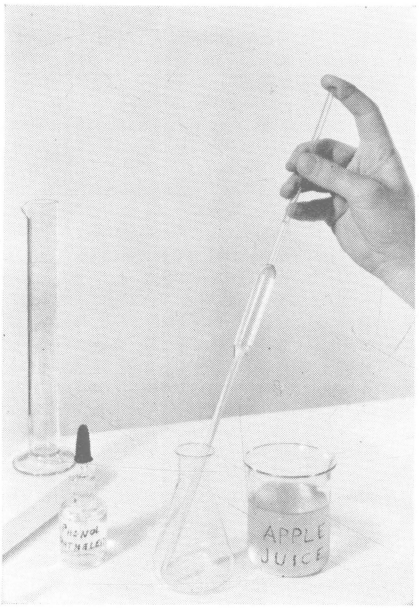

Figure 3

Fig. 3.—Move the pipette over and insert it into a flask (4). Remove the index finger from the end of the pipette and allow all of the juice to drain into the flask by gravity only.

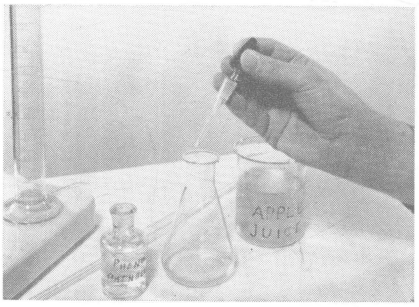

Figure 4

Fig. 4.—Add a few drops of phenolphthalein indicator to the measured sample of juice in the flask as shown in figure 4.

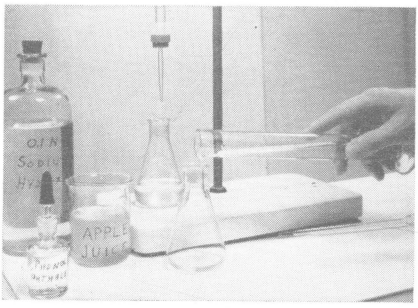

Figure 5

Fig. 5.—Add 50 ml. of distilled water to the flask as shown in figure 5. The water is measured by use of the graduate (6).

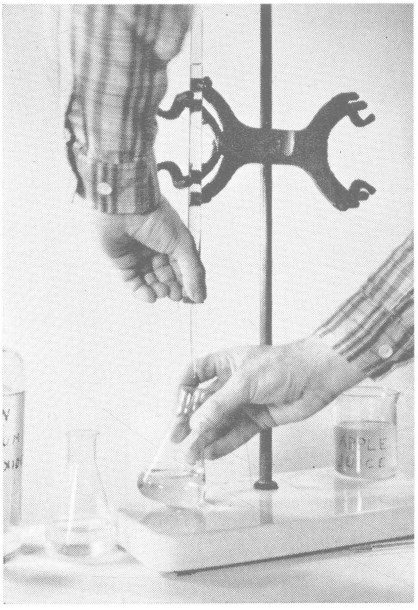

Figure 6

Fig. 6.—The level of the sodium hydroxide in the burette is read from the markings on the burette and recorded. This is the first reading. Next add the sodium hydroxide solution from the burette to the sample of apple juice in the flask until a light pink color persists as shown in figure 6. Add the sodium hydroxide slowly by turning the valve, and at the same time shake the flask with a gentle rotary motion to mix the two solution well. Reduce the rate to one drop at a time near the end until a light pink color is obtained throughout the flask. Do not add more sodium hydroxide than is necessary to produce a light pink color. A few preliminary trials may be necessary.

When the light pink color is reached, stop adding sodium hydroxide and read the level of the solution in the burette and record it. Now subtract the first reading from the second reading. This will be the total number of milliliters of 0.1 N. sodium hydroxide that was used to completely neutralize the acid in the apple juice. By referring to Table 2 the percent of acid in the apple juice can be found.

There are two flasks shown in the illustrations because the entire procedure as outlined should be done at least twice for each batch of apple juice. The two answers are then averaged.

that is added will determine the amount of neutralization that must be done. The less added sugar the more neutralization that must be done since the juice will be boiled longer and the acid will be concentrated to a greater extent. Thus the starting acidity must be lower so that the sirup will not be too tart.

The amount of sugar to add will also depend upon the sugar content of the apple juice. This can be determined by the use of a sugar hydrometer calibrated in degrees brix or calibrated as specific gravity. Table 3 shows the amount of sugar to add to an apple juice with a given percent sugar to produce a certain sugar percentage in the juice. For example, if a hydrometer reading was 12.5° brix, 397 lbs. of sugar should be added to 100 gallons of this apple juice to produce a juice with 40% sugar (Table 3).

Generally, a most satisfactory sirup can be produced by adding enough sugar to raise the sugar content to 40% or 40° Brix.

EVAPORATION—The prepared apple juice should be boiled down quickly in order to avoid too dark a product. A large low shallow pan is better than a deep narrow pan.

TABLE 2.—Weight calcium hydroxide to add to 100 gallons apple juice to reduce acidity to a given percent

Ml. 0.1N Sodium Hydroxide Required to Neutralize Acid	Percent Acid as Malic Acid	Percent Acid in Apple Juice after Partial Neutralization													
		0.15 %		0.20 %		0.25 %		0.30 %		0.35 %		0.40 %		0.45 %	
ml.	%	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.
2.0	.1340														
2.2	.1474														
2 4	.1608														
2.6	.1742		2												
2.8	.1876		3												
3.0	.2010		4												
3.2	.2144		5												
3.4	.2278		6		2										
3.6	.2412		7		3										
3.8	.2546		8		4										
4.0	.2680		9		5										
4.2	.2814		10		6		2								
4.4	.2948		11		7		3								
4.6	.3082		12		8		4								
4.8	.3216		13		9		5		2						
5.0	.3350		14		10		6		3						
5.2	.3484		15		11		7		4						
5.4	.3618	1	0		12		8		5		1				
5.6	.3752	1	1		12		10		6		2				
5.8	.3886	1	2		14		11		7		3				
6.0	.4020	1	3		15		12		8		4				
6.2	.4154	1	4	1	0		13		9		5		1		
6.4	.4288	1	5	1	2		14		10		6		2		
6.6	.4422	1	6	1	3	1	15		11		7		3		
6.8	.4556	1	7	1	4	1	0		12		8		4		
7.0	.4690	1	8	1	5	1	1		13		9		5		1
7.2	.4824	1	9	1	6	1	2		14		10		6		2
7.4	.4958	1	10	1	6	1	3		15		11		7		3
7.6	.5092	1	11	1	7	1	4	1	0		12		8		4
7.8	.5226	1	13	1	8	1	5	1	1		13		9		5
8.0	.5360	1	13	1	10	1	6	1	2		14		10		6
8.2	.5494	1	14	1	11	1	7	1	3		15		11		7
8.4	.5628	2	0	1	12	1	8	1	4	1	0		12		8
8.6	.5762	2	1	1	13	1	9	1	5	1	1		13		10
8.8	.5896	2	2	1	14	1	10	1	6	1	2		14		11
9.0	.6030	2	3	1	15	1	11	1	7	1	3	1	0		12
9.2	.6164	2	4	2	0	1	12	1	8	1	4	1	1		13
9.4	.6298	2	5	2	1	1	13	1	10	1	5	1	2		14

TABLE 2.—Weight calcium hydroxide to add to 100 gallons apple juice to reduce acidity to a given percent—Continued

Ml. 0.1N Sodium Hydroxide Required to Neutralize Acid	Percent Acid as Malic Acid	Percent Acid in Apple Juice after Partial Neutralization													
		0.15%		0.20%		0.25%		0.30%		0.35%		0.40%		0.45%	
ml.	%	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.	lbs.	oz.
9.6	.6432	2	6	2	2	1	14	1	10	1	6	1	3		15
9.8	.6566	2	7	2	3	1	15	1	11	1	7	1	4	1	0
10.0	.6700	2	8	2	4	2	0	1	12	1	8	1	5	1	1
10.2	.6834	2	9	2	5	2	1	1	13	1	10	1	6	1	2
10.4	.6968	2	10	2	6	2	2	1	14	1	11	1	7	1	3
10.6	.7102	2	11	2	7	2	3	1	15	1	11	1	8	1	4
10.8	.7236	2	12	2	8	2	4	2	0	1	12	1	9	1	5
11.0	.7370	2	13	2	9	2	5	1	1	1	13	1	10	1	6
11.2	.7504	2	14	2	10	2	6	2	2	1	14	1	11	1	7
11.4	.7638	2	15	2	11	2	7	2	3	2	0	1	12	1	8
11.6	.7772	3	0	2	12	2	8	2	4	2	1	1	13	1	9
11.8	.7906	2	1	1	13	2	9	2	5	2	2	1	14	1	10
12.0	.8040	3	2	2	14	2	10	2	6	2	3	1	15	1	11
12.2	.8174	3	3	2	15	1	11	2	7	2	4	2	0	1	12
12.4	.8308	3	4	3	0	2	12	2	8	2	5	2	1	1	13
12.6	.8442	3	5	3	1	2	13	2	10	2	6	2	2	1	14
12.8	.8576	3	6	3	2	2	14	2	11	2	7	2	3	2	15
13.0	.8710	3	7	3	3	3	0	2	12	2	8	2	4	2	0
13.2	.8844	3	8	3	4	3	1	2	13	2	9	2	5	2	1
13.4	.8978	3	10	3	5	3	2	2	14	2	10	2	6	2	2

The end point of evaporation is when the sirup reaches a sugar content of about 70% or 70° brix. This can be measured by a refractometer, a sirup hydrometer, or a thermometer. The refractometer is quite expensive and not necessary unless very accurate measurements are desired. A sirup hydrometer such as is used for maple sirup can be used satisfactorily. The best and easiest method is the use of an accurate and easily read thermometer. The eye should always be exactly at the level of the column of mercury when a reading is taken.

The boiling point for an apple juice-sugar solution of 70% is 223.5° F. at sea level. Factors which will affect this figure are altitude, weather, and accuracy of the thermometer. These factors can be eliminated by taking a reading with the thermometer in boiling water just

TABLE 3.—Pounds of sugar to add to 100 gallons of apple juice to bring the sugar level up to a given percent

Degrees Brix or % Sugar in Apple Juice	Specific Gravity of Apple Juice at 68° F.	Percent Sugar in Apple Juice after Addition of Sugar					
		20%	25%	30%	35%	40%	45%
10.0	1.03998	108	173	248	333	433	552
10.5	1.04206	103	168	241	327	426	544
11.0	1.04413	97	162	235	320	419	536
11.5	1.04622	92	156	229	313	412	528
12.0	1.04831	87	150	223	307	405	520
12.5	1.05041	81	144	217	300	397	512
13.0	1.05252	76	139	211	293	390	504
13.5	1.05464	70	133	204	287	383	497
14.0	1.05677	65	127	198	280	376	489
14.5	1.05890	60	121	192	273	368	481
15.0	1.06104	54	116	186	267	361	473

before using it in the sirup. To the reading obtained in boiling water, add 11.5 which is the difference between 212° F. and 223.5° F. This new figure is the end point for the sirup. For example, if the reading in boiling water is 210.5° F. and 11.5 is added to this figure, the result is 222° F. This temperature of 222° F. is the end point temperature for a sirup of 70% sugar.

BOTTLING—The sirup should be filled while hot into suitable containers. If the containers are small, one quart or less, they can be filled immediately when the sirup is finished. If large containers are to be used, the sirup should be cooled by a milk cooler or some similar cooler to about 180° or 190° F. before filling. The temperature of 180° F. will kill any molds in the container and cap and thus prevent molds from forming on top of the sirup. If large containers are filled too hot, they will remain hot too long and produce too dark a sirup and cause a reduction in flavor. The containers as soon as filled should be turned on their side or upside down for several minutes to kill the molds on the caps.

LABELING—The Division of Food and Dairies of the Ohio Department of Agriculture in consultation with Federal Food and Drug Administration officials have interpreted the Ohio Food, Drug, Cosmetic, and Device Law in essence as follows:

(1) Only that sirup made from apple juice without added sugar is entitled to bear the name "Apple Sirup". In addition, the phrase "made from depectinized, partially neutralized, filtered apple juice" must appear on the label.

(2) Sirup made from apple juice and added sugar where more than 50% of the total sugar content is contributed by the apple juice shall be labeled "Apple and Sugar Sirup". In addition the phrase "made from depectinized, partially neutralized, filtered apple juice and sugar" must appear on the label.

(3) Sirup made from apple juice and added sugar where less than 50% of the total sugar content is contributed by the apple juice shall be labeled "Sugar and Apple Sirup". In addition the phrase "made from depectinized, partially neutralized, filtered apple juice and sugar" must appear on the label.